



**UNITED STATES ENVIRONMENTAL PROTECTION AGENCY**  
**REGION 10**  
1200 Sixth Avenue  
Seattle, WA 98101

November 14, 2002

Reply To  
Attn Of: ECL-113

Ms. Kathleen Hain, Manager  
Environmental Restoration Program  
U.S. Department of Energy  
Idaho Operations Office  
850 Energy Drive  
Idaho Falls, Idaho 83401-1563

Re: EPA Review of the 2002 draft Remedial Design/Remedial Action Work Plan and Field Sampling Plan for the Operable Unit 4-13, CFA-04 Pond

Dear Ms. Hain,

EPA received the RD/RA Work Plan and FSP on September 30, 2002. EPA comments on these documents are enclosed. Important issues that need to be resolved in the work plan include demonstrating appropriate waste characterization of soil for disposal at the ICDF and verifying the quality of the methyl mercury analysis in support of the revised final remediation goal for mercury.

I look forward to resolving these issues during the comments resolution period. Please give me a call at (206) 553-0040 if you have any questions.

Sincerely,

  
Kathy Ivy  
Remedial Project Manager

Enclosure

cc: Carol Hathaway, DOE-ID  
Clyde Cody, IDEQ

**EPA November 2002 Comments on  
the Remedial Design/Remedial Action Work Plan and  
the Field Sampling Plan for the Operable Unit 4-13 CFA-04 Pond**

**General Comments**

1. The Pre-Remediation Sampling Summary Report included in Appendix D of the RD/RA Work Plan does not include a sufficiently detailed description of the methyl mercury analysis to support an evaluation of the methyl mercury sampling results. A copy of the EPA comments on the methyl mercury analysis in response to the pre-remediation sampling plan is attached. The pre-remediation sampling report should address these comments as well as the following additional questions: First, information provided following submittal of the RD/RA Work Plan described poor matrix spike recovery for methyl mercury. What is the required matrix spike concentration for a 75%-125% recovery and what concentration was used as part of the methyl mercury analysis? Also, was methyl mercury or simply mercury used as the spike? Second, a suggestion was made following submittal of the RD/RA Work Plan to use the reported values of methyl mercury adjusted for the poor spike recovery rather than using the method detection limit of 0.005 mg/kg to derive the percentage of methyl mercury in the soil. Did the laboratory run an extended calibration curve to determine if results below the method detection limit are linear? Also, what were the methyl mercury concentrations detected in the blanks? The potential for accumulating error while calculating the percentage of methyl mercury should be evaluated and the associated level of confidence in the calculated FRG of 8.4 mg/kg should be discussed. (K.I.)
2. The description of soil removal, segregation, treatment, field testing and other sampling is not sufficiently detailed for purposes of disposal at the ICDF. Section 5 of the RD/RA Work Plan states that soil removal will be primarily based on the results of pre-remediation sampling and will follow drawings in Appendix A and specifications in Appendix B. (1) Concerning soil removal, segregation, and treatment: (a) More information should be provided to explain how each waste stream will be separated and accumulated as each zone is excavated based on the waste streams identified from pre-remediation sampling. (b) Information should be provided in the text about contamination found in Zones 6A and 7A and in areas where miscellaneous sampling was performed as described in the Pre-Remediation Sampling Summary Report and plans for management of these waste streams should be discussed. (c) The process for treating TCLP mercury contaminated soil should be described in some detail. (2) Concerning field testing and other sampling: (a) Various sections in the RD/RA Work Plan discuss field screening of soil, but it is not clear if field screening will occur during excavation to help characterize the soil for disposal and/or if field screening will commence after known depths of contamination have been reached to determine if additional soil removal is necessary. If field screening will be used to help characterize the soil for disposal, information about the screening instrument should be provided and the quality of data generated from this instrument should be demonstrated. (b) If pre-remediation sampling results alone are expected to be sufficient for purposes of disposal at the ICDF, a statistical analysis needs to be performed to demonstrate that there is sufficient probability that the soils will not exceed the ICDF WAC. If additional sampling of excavated soil is necessary to ensure that the ICDF disposal criteria have been met, plans for this sampling effort

**7. Page 3-2, Section 3.2, last paragraph in section:** An explanation should be provided for the increase in the estimated volume of contaminated soil from the value included in the RI/FS to the value calculated following pre-remediation sampling. (K.I.)

**8. Page 3-2, Section 3.3:** Site preparation also includes completion of the waste profile information to support shipment of the wastes to the ICDF. (W.P.)

**9. Page 4-2, Table 4-1, "Toxic air emissions" and "Emission monitoring":** The compliance strategy column for the ARAR "Toxic air emissions" states that monitoring will not be required because air emissions modeling indicates that contaminant levels will be below regulated levels. The compliance strategy column for the ARAR "Emission monitoring" implies that air emissions monitoring may be required. This inconsistency needs to be resolved. (K.I.)

**10. Page 4-2, Table 4-1, "Emission monitoring" and "Hazardous Waste Determination":** The compliance strategy columns for these ARARs refer to the "drain field site" and the "engineering cap," respectively. This information needs to be updated to reflect remediation at CFA-04. (K.I.)

**11. Page 5-2, Section 5.3.3, second paragraph, last sentence:** The possible disposal facilities for contaminated posts should be identified. (K.I.)

**12. Page 5-2, Section 5.3.3, third paragraph, last sentence:** A survey for radiological contamination is described for the power pole, but no mention is made of testing for mercury. (K.I.)

**13. Page 5-3, Section 5.3.6, first and second paragraph:** The RRWAC and other INEEL documents are referenced here for management of ACM. A brief summary of the requirements in these documents should be included in the RD/RA Work Plan. (K.I.)

**14. Page 5-4, Section 5.3.6, paragraph a top of page:** It states here that "appropriate controls" will be implemented if non-roofing ACM is encountered. A brief description of these controls should be included. (K.I.)

**15. Page 5-4, Section 5.3.6, last paragraph in section:** It is not clear if the ACM that was sampled in 1994 was primarily roofing material or if the samples of ACM were commingled with soil. If these samples consisted primarily of roofing material, this does not provide assurance that an any ACM/soil mixture that might be discovered during excavation does not contain mercury above the FRG. (K.I.)

**16. Page 5-4, Section 5.3.7:** Although 4 categories are established for the contaminated soils, nothing is mentioned concerning the confidence level of each assumption, which is necessary. Sampling should be proposed during the excavation to safeguard that soils in each category are within specification and will not fail verification at the ICDF. (W.P.)

**26. Appendix B, Page 5, Line 6:** It states here that excavations will be limited to 10 feet below the ground surface. If contamination is found deeper than 10 feet, it would be prudent to consider removing contaminated soil to greater depths in order to avoid placing long-term institutional controls at the site. (K.I.)

**27. Appendix B, "Earthwork," Page 4, Line 6 through 18:** This section of the text describes the use of liners to transport the excavated soil. However, the text does not include details of how the soils will be moved from the excavation, placed in the liners or how the liners will be designated for identification during shipping and disposal. Please include additional details to the text describing these activities. (J.R.)

**28. Appendix B, "Earthwork," Page 6, Line 6, last sentence:** The time provided for the return of the soil confirmation sampling is stated as 5 weeks in this portion of the text. If the remediation goals are not met in a particular portion of the excavation the contaminated residual soil will be exposed to wind for over a month. A shorter turnaround time and the use of tarps as temporary cover is recommended to minimize the potential for windblown movement of contaminants at the excavation site. (J.R.)

**29. Appendix B, "Earthwork," Page 6, Line 14, first paragraph:** This portion of text states that laboratory bottles are present as debris on the CFA-04 site. The text should include details of the procedures that will be followed to collect and analyze any liquid residues that may be found in the glassware. If residual liquids are found in bottles they will require different handling and disposal than the soils. (J.R.)

**30. Appendix B, "Earthwork," Page 6, Line 33, Soil Removal from the Basalt Surfaces:** This section states that the subcontractor will make every effort to remove contaminated soil from the basalt surfaces. Specific details of how the vacuuming will be performed and what will constitute sufficient removal effort are not provided. Please provide the details of how this portion of the soil removal activities will be performed and a clear criteria for determining when removal activities should be concluded. (J.R.) Also, this information should be included in the body of the RD/RA Work Plan in Section 5.3.7. (K.I.)

**31. Appendix B, "Earthwork," Page 7, Line 7 through 21:** It should be demonstrated that the pit run gravel backfill plus 6 inches of material pulled from the surroundings will be sufficient to allow for revegetation. Six inches of topsoil does not seem sufficient when compared with the requirements listed in the construction specifications for "Revegetation," Page 3, Line 11 where it states that soil will be tilled to a depth of 4 inches with a firm seedbed below the seeding depth. (K.I.)

**32. Appendix D, Page D-13, Section 2.3, second paragraph, last sentence:** A summary should be added at the end of this report to address the question about chromium and silver. As it is, information about whether the soil is characteristic for these metals can only be found by looking through the results of every sample in each zone. (K.I.)

**42. Field Sampling Plan, Page 3-5, Section 3.1.7.1, third paragraph:** Please explicitly specify whether field screening data or laboratory analysis will be used to analyze the initial set of confirmation samples; the analysis method can be a significant source of variance in the data set, and it is recommended that the results of laboratory (as opposed to field) analysis be used to estimate the variance. If this is not to be the case, then please provide data justifying the use of a variance calculated from field analyzed data (i.e., data showing that the variance estimated by the two analytic methods are comparable). (L.W.)

**43. Field Sampling Plan, Page 3-5, Section 3.1.7.1, third paragraph:** An alternative, and possibly more efficient, method to develop an estimate of the data variance is contained in Section 6.3.1.2 (page 6-5) of EPA, February 1989, *Methods for Evaluating the Attainment of Cleanup Standards, Volume 1: Soils and Solid Media*, EPA 230/02-89-042 (EPA 1989). This method recommends analyzing 20 random samples in order to estimate sample variance. If the resulting variance indicates less than 20 samples are required, then the mean and UCL can be calculated from the existing samples. This does, of course, assume that the 20 samples would be subjected to a laboratory analysis. (L.W.)

**44. Field Sampling Plan, Page 3-5, Section 3.1.7.1, fourth paragraph and ensuing formulae:** If the distribution is determined to be log-normal, and the number of samples is less than twenty, then it is strongly recommended that alternate calculations, appropriate for the log-normal, be used for the mean and UCL (see, for example, methods presented in Chapter 13 of *Statistical Methods for Environmental Pollution Monitoring*, Richard O. Gilbert, 1987). Also, if the data is log-normal, note that EPA guidance requires that the log-normal (transformed) UCL be compared to the transformed (log) cleanup standard, and specifies that the log-normal UCL should not be transformed back and then compared to the untransformed cleanup standard (see last paragraph of Section 7.6 [page 7-21] of EPA 1989). Recommend adding a discussion of calculation methods to be used in the case of a log-normal distribution. (L.W.)

**45. Field Sampling Plan, Page 4-1, Section 4.3.1, Second Paragraph:** In reference to confirmation sample locations, this section states that, "The potential sampling points were only selected from those areas of the pond that had been excavated during the remediation effort." This statement is unclear. According to the text, this pond was previously excavated during a time critical action and will be further excavated in order to meet the remedial goal for mercury. Please explain why confirmation samples of a future excavation, that one that will remove mercury contaminated soils below the remedial goal, would be based on areas removed during a previous excavation? Confirmation samples should be collected throughout the entire pond area that was excavated during both removal actions in order to confirm that mercury is indeed below remedial goals. Please clarify the text. (A.P.)

Also, confirmation samples appear to be planned for only the bottom surface of the pond. Because some zones will be excavated to greater depths than surrounding zones, samples should be taken from the vertical surface of deep excavations to ensure that the contamination has been chased to its horizontal extent. (K.I.)

16 October 1999

Reply to  
Attn of: OEA-095

Subject: QA Comments on Methyl-Mercury Analysis Information Proposed by INEEL

From: Bruce A. Woods, Ph.D.  
QA Team Leader, Chemist, CLP TPO  
Quality Assurance, Monitoring, & Assessment Unit

To: Kathy Ivy  
Superfund Program

The Quality Assurance Unit has completed its review of the document proposed methyl-mercury analysis information from INEEL. In an e-mail from Carol Hathaway at INEEL to you, were mentioned the following articles or methods:

Determination of Methylmercury in Sediments by Steam Distillation/ Aqueous-Phase Ethylation and Atomic Fluorescence Spectrometry, K. C. Bowles and S. C. Apte, Analytical Chimica Acta, Vol. 419 (2000), pp. 145-151.

Determination of Methylmercury in Natural Water Samples by Steam Distillation and Gas Chromatography-Atomic Fluorescence Spectrometry, K. C. Bowles and S. C. Apte, Analytical Chemistry, Vol. 70(1998), pp. 395-399.

EPA Method 1630 - Methyl Mercury in Water by Distillation, Aqueous Ethylation, Purge and Trap, and CVAFS

At the present time, I have only received from our library the Analytica Chimica Acta paper, and was able to locate EPA Method 1630 on the web. I am still waiting for the Analytical Chemistry paper, but I doubt that it will change the comments provided below.

Comments on Analytica Chimica Acta paper

1. This paper describes the optimization of the steam distillation process and the reagents used for this experimental procedure, such as quantity and concentration of acids added, quantity of copper salt added to enhance recovery, etc. Because the e-mail message only briefly described the general process that would be used for analyses using information from the two published papers cited above and EPA Method 1630, INEEL must have a

written and document-controlled Standard Operating Procedure that provides all of the analytical handling of the sample through the analytical process. This must be in similar level of detail to the details presented in EPA Method 1630. If the results of these analyses are included in any reports or database systems, the results must be referenced back to the Standard Operating Procedure followed to generate the results. Preparation of written and document-controlled Standard Operating Procedures is a routine QA practice. By looking over these papers and EPA Method 1630, specific details for the analyses of samples for methylmercury cannot be determined at this time or for similar analyses that may be conducted in the future on other sites or for comparison over time.

2. It was noted in the paper (p. 149) that if high concentrations of inorganic mercury were present in the sample, that diethylmercury could overlap the peak for methylethylmercury. What QC checks has INEEL implemented to determine if this is a problem with the samples to be analyzed for this project?
3. It was noted in the paper (p. 145) that a highly sulfidic sediment sample had much lower recovery (76%) compared to the other samples (>95% recovery). Will the presence of sulfidic sediment be a concern for this project or will such a low recovery be of concern for the planned data collection activity? What QC checks will be implemented to determine if this is a concern for this project?

#### EPA Method 1630

4. We found the January 2001 version of this method on the Internet on a non-EPA site. It is labeled as a DRAFT method. We have no idea how much round robin evaluation, if any, this method has undergone to determine reproducibility or robustness by labs other than the lab that developed this method.
5. This method specifies careful distillation (Section 4.4.2) and control of the amount of acid added to the samples which could interfere (low or high bias of the data) with the later analysis of the sample. Because of the lack of specific details on the INEEL procedure, we do not know if this will be a concern during analysis of samples for this project.
6. The method notes (Section 4.4.3) that samples preserved with nitric acid (most commonly used for metal sample preservation) can have degradation of methylmercury occur during the sample distillation step. What preservation will INEEL be using?
7. We assume that INEEL will be running all of the laboratory quality control samples specified in Section 9.0 of this method, ie., MS/MSD, three method blanks per batch, IPR and OPR, etc., at the frequency specified in the method or at a higher frequency because this method is being adapted to a matrix for which the method has not been validated for (running method on soil but method applies to water samples).

8. INEEL should also discuss what types of field QC samples will be collected, such as field replicates, field blanks, etc., and at what frequency these field QC checks will be collected. These are not necessarily specified in the method.

If you wish to discuss the contents of this memo, please feel free to contact me at 206 553-1193.